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I.G. FARBENINDUSTRIE A.G.
LEUNA, GERMANY

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COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE

LONDON—H.M. STATIONERY OFFICE

I.G. FARBENINDUSTRIE AG
LEUNA, GERMANY

10 May, 1945.

Reported by:

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CIOS BLACK LIST ITEM - 22
Miscellaneous Chemicals

COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE
G-2 DIVISION, SHAEF (REAR) APO 413

TABLE OF CONTENTS

Subject	Page No
1. Introduction	3
2. Processes	
a. Chief Organic Compounds	3
b. Luran	3
c. Triethanolamine	3
d. Glycerine Substitute	4
e. Phosgene	4
f. Oxidation of Hydrocarbons	4
g. Soap Substitute	4
h. Aldehydes	6
i. Fatty Acids	6
j. Synthesis of Benzene	6
k. Alanine	6
l. Aliphatic Amines	6
m. Adipic Acid	6
n. Resin	7
o. Production Figures	7
3. Research Programme -- Organic	8

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I.G. FARBENINDUSTRIE AG

LEUNA, GERMANY

1. INTRODUCTION

This plant was visited on 10 May 1945. It has been very badly damaged by bombing, although it is estimated to have remaining 15% of its synthetic ammonia capacity or 150 T/day. Information on organic products of this plant was supplied by Dr. Johann Giesen, Director in charge of organic work at Leuna.

2. PROCESSES

a. The chief organic compounds made at this plant were as follows:

Methanol, isobutanol, and higher alcohols
Lubricating oils (from ethylene)
Phenol, cresols, xylenols, pyrocatechol, and resorcinol
Cyclohexanol and cyclohexanone
Epsilon amino caproic acid (an intermediate for "Luran", the IG "Nylon")
Adipic acid
Alcohols containing up to 10 carbon atoms, with traces of C_{12} alcohols
Tri-hydroxymethyl ethane
Iso octane
Alkylate (Referred to as ET 120)
Mersol and Mersolat (soap substitutes, made from a Fischer-Tropsch oil fraction)
Aldehydes (e.g, Iso-butyric Aldehyde)
Esters from adipic acid and Leuna alcohols
Acids from the alcohols
Mono-, di-, and tri- methyl amines

b. Luran was not made at Leuna, the intermediates being shipped to other IG plants at Landsburg, Berlin, Premnitx, and Wolfen.

c. Triethanolamine was made by IG at Ludwigshafen, but not at Leuna.

d. A glycerine substitute was made here, by condensing form-aldehyde with propionaldehyde in alcoholic solution in the presence of calcium oxide.

$$CH_3\text{-}CH_2\text{-}CHO + 2HCHO \xrightarrow{CaO} CH_3C(CH_2OH)_3$$

The reaction was carried out at 30-40°C by merely mixing the reagents. It is very rapid and strongly exothermic, so that cooling is necessary. The product was filtered off, washed with i-butyl ketone, and crystallized from water. It was used chiefly as a glycerine substitute in making the alkyd type of resin, under the name P-3.

e. The Leuna plant made no phosgene, diphosgene, or other war gases.

f. The Leuna plant did no work on the oxidation of hydro-carbons.

g. One of the important products made at Leuna was a soap substitute, or soap extender. This was made from a special fraction of Fischer-Tropsch oil known as Kogasin, which was the fraction having a distillation range of 230-330°C and an average chain length of 15 carbon atoms. It was a straight chain aliphatic compound, with no branching. As received, it contained both oxygen derivatives and unsaturated compounds, which were eliminated by catalytic hydrogenation, using a nickel tungstate catalyst, a pressure of 200 at. and a temperature of 300-350°C. By this procedure was obtained a mixture of straight chain hydrocarbons, containing the compounds from $C_{12}H_{26}$ to $C_{18}H_{38}$, but chiefly $C_{15}H_{32}$. This mixture is then treated with sulphur dioxide and chlorine, while being irradiated with "ultra violet" light. The temperature is held at 30-40°C by means of an external pipe-type heat exchanger.

The reaction is as follows:

$$CnH_{2n+2} + SO_2 + Cl_2 \xrightarrow{\text{light}} C_nH_{2n+1}SO_2Cl + HCl$$

and as secondary reaction

$$C_nH_{2n+1}SO_2Cl + SO_2 + Cl_2 \xrightarrow{\text{light}} C_nH_{2n}(SO_2Cl)_2 + HCl$$

The reaction is carried to only 70% of completion, as too much disulphonyl chloride is formed if it is attempted to react more than this percentage of the oil. The product was, for the most part, shipped to soap makers (who saponified it together with their other soap making materials).

A cresol ester was also prepared from this sulphonyl chloride and used to plasticize polyvinyl chloride. Also, a small amount of the sodium salt (Mersolat) was made by saponifying the Mersol at 70°C with 10% NaOH solution. The resulting solution of sodium alkyl sulphonate was allowed to stand till most of the oil separated, the clear aqueous layer being then drawn off and dried on a drum drier. About 5% of the oil remained in solution in the aqueous layer, and was steam distilled off during the drum drying operation. The recovered oil contained chlorine derivatives, and was further chlorinated and sold for use as a solvent.

The finished sodium alkylsulphonate was sent to Schkopau for use in the preparation of polyvinyl chloride emulsions. According to Dr. Giesen, the Mersolat should not be considered as a soap but a solvent, but which it is believed would be more accurately referred to as a dispersing agent. The equipment in which the production of the sulphonyl chloride was carried out consisted of 20 vertical steel towers, each approximately 20 feet high by 8 feet inside diameter; the bottom or reacting section of each of these containing 16 glass tubes 7 inches inside diameter running across the steel cylinder in a horizontal position. In each of these there were placed six 40-60W daylight lamps; that is, the type which start on a tungsten filament and then operate as a mercury vapor light. In order to transmit as much ultra violet light as practicable, the glass tubes were made of "Normal" glass. The reacting section was 8 feet high. The entire steel cylinder and all pipe lines were lined with an acid resistant resin of the "Haveg" type which lasts from six months to one year. The upper part of the reactor was filled with rings of this same resin. In operating, the Kogasin was circulated from the bottom of the reactor through a heat exchanger consisting of water cooled six inch pipe also lined with "Haveg" and was returned to the top of the tower. Chlorine and sulphur dioxide were passed in at the bottom of the reactor and the hydrochloric acid, mixed with some excess sulphur dioxide, passed off at the top. Circulation was brought about by means of a centrifugal pump with a porcelain impeller, and the valves were steel lined with "Haveg". The lining of the towers had at first been done by using thin sheets of resin, cemented in place, but this had not been found satisfactory so that they had switched to the use of a solution applied by spraying while the equipment was heated to a temperature of 100-200°C. This latter procedure had been found quite satisfactory. At Leuna they had made 4500 tons (metric) per month of Mersol prior to the damaging of the plant by bombs. This would give a capacity of 225 tons per month per tower. The product as made contained 30% unreacted oil and about 70% of sulphonyl chlorides. Of the 70%, 50-60% was mono-sulphonyl, the remainder being a mixture of di-and higher substitution products which are useless in the soap which is made from the Mersol.

h. Aldehydes were made at the Leuna plant from the alcohols produced there.

i. Fatty acids were made at this plant from the highest fractions of the alcohols remaining from iso-butyl alcohol manufacture by caustic fusion and precipitation with sulphuric acid. Some of these fatty acids were esterified with trimethylolethane to form a castor oil substitute for use as a plasticizer. The remainder was shipped to the IG plant at Griesheim, where the cobalt and manganese salts were prepared. These salts were used as driers under the trade name of "Solagen".

j. Contrary to one report received, there had been no work done of the synthesis of benzene at Leuna. The work referred to was due to a misunderstanding; it had been directed toward the production of benzine, or motor gasoline.

k. At the Leuna plant alanine (1 aminopropionic acid) which they termed "alkazidlauge" is used for the removal of carbon dioxide from process gases. This solution absorbs CO_2 when cool and under pressure, and releases it when heated without pressure.

l. The alophatic amines were made by the usual procedure of reacting the alcohols with ammonia in the presence of a catalyst, and were shipped to other IG plants, chiefly to Ludwigshafen, for further processing.

m. Adipic acid was made by the route phenol-cyclohexanol-cyclohexanone-cyclohexanone oxime-caprolactam-adipic acid. This route was preferred to a simpler one as it gave more by-products and intermediate products which could be used as the raw materials for other syntheses.

n. The only resin or plastic made at this plant was of the phenol-formaldehyde type (Mepasin).

o. The production figures for January, 1944 are an approximate measure of the capacity of this plant before it was bombed; and the organic products are listed in the following table.

Product	Production Metric Tons	Product	Production Metric Tons
Crude methanol	15850	x3 SS oil	728.1
Crude i-butanol	14690	Spindle (Achsen)oil	117.8
Refined Methanol	11287	Cuttingoil	28.1++
Anhydrous "	487	Machine oil	77.1
Dimethyl ether	75.8	Hydraulic oil	32.5
Propanol	302.9	Mesamoll	359.8
Dipropyl ether	.4++	Weichmacher	
Crude i-butanol	1137.6	Thiocompounds	2.7
Refined "	848.5	Methylamines	58.0
Pentanol	216.2	i-Butylamine	1.6
Ketoalcohols	37.5	Other amines	12.6
Other alcohols	66.7	Catalysts	277.1
Intrasolvane (Butanol-Pent-anol)	638.3	Mepasin	4300.0
		Mersol,D,H,30	4868
Formaldehyde	114.9	Mersolat & Mersapon	89
Propionaldehyde	65.2	Hydrochloric acid	1405
Other aldehydes	.6++	Ethyl chloride	159
Trimethylolethane	60.0	Dichloroethane	12
Ketone oil	179.8	Carbolic oil	641
Carbon Dioxide	111.3	Phenol pure & crude	842
i-Butylene	32.9	Cresols	498
i-Octane	488.7	Xylenols	287
Alkylate (ET 120)	1693.7	Crude catechol	250
x1 Arobin	55.1	Tech "	5
HS olefin & polymers	51.8	Oxime (cyclohexanone)	240
x2 Noral	11.6++	Lactam(")	155
		Adipic Acid	95

++ February figures. No January production.

x1-The last fraction of the catalytic cracking process, hydrogenated.

x2-A special fraction used as a solvent for insecticides.

x3-A polymerized ethylene lubricating oil. Two grades SS 903 and SS906 were made, with Engler viscosities at 100°C of 3 & 6 respectively.

3. RESEARCH PROGRAMME - ORGANIC

This field of investigation was the province of Dr.Herold, who, under Dr.Giesen, was responsible for the following general programme;

1. Devolopment of new uses for high molecular weight alcohols.
2. Finding uses for phenols, cyclohexanol, and cyclohexanone.
3. Development of new catalysts for the synthesis of i-butylene.
4. Improving processes for the extraction of phenols.
5. Improved methods for the manufacture of maleic anhydride, adipic acid, and other intermediates for plastics.
6. Development of new catalysts for alcohol syntheses.

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